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TESTS ON "COLD FUSION" IN AN ELECTROCHEMICAL CELL

Report No 90-28-K

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ABSTRACT

Results of preliminary tests on "cold fusion" in an electrochemical cell are reported. Tests were carried out on palladium rod and disc cathodes with 0.1M LiOD in D_2O electrolyte. Several treatments were carried out on the cathodes including heat treatment in a vacuum, melting, mechanically deforming, palladizing, running at high current density, cycling the current from low to high values and poisoning the cathode surface. No excess heat, tritium and gamma activity were found in these tests.

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EXECUTIVE SUMMARY

TESTS ON "COLD FUSION" IN AN ELECTROCHEMICAL CELL

Considerable controversy has followed the claim made by Fleischmann and Pons of fusion at low temperature in an electrochemical cell consisting of a palladium cathode and an electrolyte of lithium deuteroxide in heavy water. The potential of this technique for electricity generation resulted in the initiation of preliminary laboratory tests which were performed with many modifications as new information became available.

The major findings reported in the literature were (i) the inability to reproduce the results by highly respected institutions with qualified personnel, sophisticated equipment and comprehensive test programs, (ii) the production of large quantities of tritium by a few laboratories but in only a fraction of the cells tested in each laboratory, (iii) variable production of neutrons from zero to a few standard deviations above background and (iv) the production of zero to 20% excess heat. The initial belief in the scientific community was that the composition of the palladium cathode and its preparation was an important factor and a D/Pd ratio in excess of unity was necessary. However, we believe that methods used in measuring this ratio were unsatisfactory.

Several rod (0.1 to 0.6 cm dia) and disc shaped electrodes (~1 cm dia x 0.2 cm thick) were investigated in this work. A variety of palladium pretreatments were carried out including heat treatment under vacuum to remove residual hydrogen with slow cooling to maintain relatively large grains, melting several times to minimize impurities and mechanical deformation to harden the cathode.

Reliable data were obtained with the inclusion of a heater in one cell for calibration and heat balance purposes, the recognition of the possibility of chemiluminescence in tritium measurements, and the allowance for variable background counts during the gamma and neutron emission measurements.

No excess heat, tritium, gamma-ray or neutron activity were found in these tests although a variety of techniques were tried. These included reversing the current to remove impurities, palladizing the cathode, adding other cations to grow dendrites on the cathode surface, adding poisons to inhibit recombination of D atoms to D_2, increasing the current density and cycling the current between high and low values. In one electrode, we achieved a D/Pd ratio of 0.78, determined by heating and gas collection.

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To

Dr. D. Mills
Director of Research

TESTS ON COLD FUSION IN AN ELECTROCHEMICAL CELL

1.0 INTRODUCTION

The achievement of fusion at low temperature in an electrochemical cell has recently been claimed by Fleischmann and Pons(1989). The potential of this technique for electricity generation resulted in the initiation of laboratory tests. These were hastily put together and performed with many modifications as new information became available to determine the excess heat generation capability. The results of these preliminary tests are reported here.

From the few reports of "success" in the scientific community, the significant findings are:

(a) Several laboratories have reported the production of tritium. It appears in less than 25% of the cells tested by each laboratory. Dendrites are reported present on electrodes in cells producing tritium. The high electric fields at the tips of dendrites may be responsible for high acceleration of D nuclei causing fusion but it is thought that distances are inadequate for high velocities to be obtained.

(b) There appears to be conflicting information as to the necessity of pretreatment of the palladium cathode. According to one laboratory (Murphy, Texas A.& M.), off the shelf palladium wires produced excess heat while another laboratory (Huggins, Stanford) carried out many meltings of the palladium followed by mechanical deformation.

(c) There also appears to be conflicting information as to the time required to precharge the electrode at low current density. Stanford reported excess heat immediately after a cell is turned on while Texas A. & M. used a two day precharge time for heat measurements and as long as fourteen days with 1 mm dia electrodes increasing to 28 days for 3 mm dia electrodes when carrying out tritium measurements.

(d) The production of neutrons on fracturing of lithium deuteride crystals has been reported by Klyuev et al(1986) and may be a possible mechanism for the cold fusion observations.

2.0 EXPERIMENTAL DETAILS

2.1 Palladium Rod Electrode Experiments

A 200 mL Pyrex glass cell (cell 1, Figure 1) was set up with a palladium rod cathode, a platinum cage anode and initially an electrolyte consisting of 0.1 molar lithium deuteroxide in heavy water. Other smaller cells (cells 2,3,and 4) were also investigated in parallel as described in Table 1 and Appendix A.

2.1.1 Rod Cathode

The cathode was 0.6 cm in diameter and 7 cm long. It was prepared by cutting a strip from a 10 oz slab of palladium from Johnson Matthey and machining into a cylindrical rod. The claimed purity of the palladium was 99.9% but it is believed that this refers only to other metallics present and not to other impurities not analysed such as graphite. Stainless steel wire was then silver soldered to one end of the palladium. Spot welding of platinum or a mechanical connection will be considered in the future since the solder was later found to contain significant amounts of copper and zinc. The palladium was then heat treated at 800°C for 18 hours in a low vacuum to remove hydrogen and then cooled slowly. It is believed that large grains are formed under these conditions. Present knowledge indicates that heat treatment in argon or under a small pressure of deuterium is preferred to exclude oxygen. The rod was then degreased in acetone and a glass tube was cemented with epoxy over the stainless wire to isolate the wire from the electrolyte which covered all of the palladium.

2.1.2 Heat Measurements on Rod Cathode

A small electric cartridge heater with an inconel sheath was included in the cell for determining the thermal capacity of the cell. A fan was also added to allow operation at low cell temperatures above ambient and minimize extrapolation backwards to the y-axis in the determination of excess heat. High current densities required the cell be immersed in a bath through which water was continuously recirculated. For safety reasons, the current to the cell was made interruptible by a temperature switch set at about 90°C.

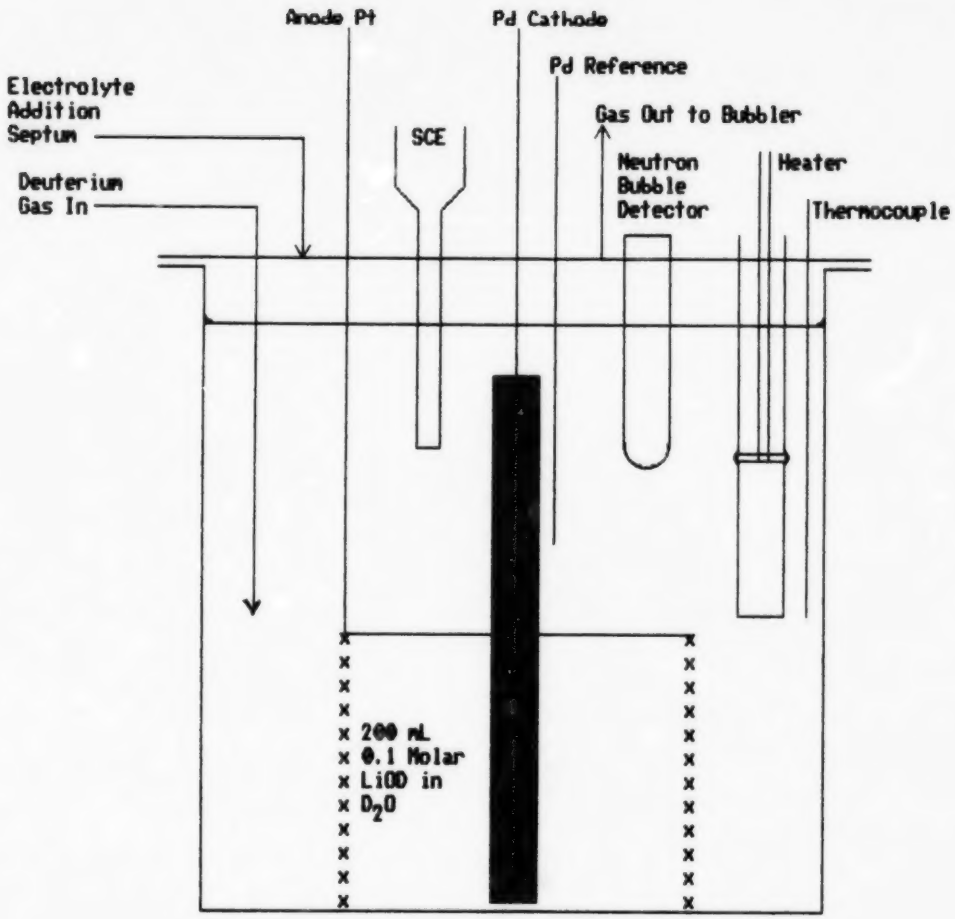

Figure 1 Schematic of Electrolytic Cell

TABLE 1

SUMMARY OF TEST CELLS FOR RODS

Cell Number	Cathode	Anode	Cell Volume (mL)	Electrolyte
1	Pd rod 0.6 cm dia, 7 cm long machined	Pt mesh 3.5 cm dia 2 cm high 66 sq cm area	150	0.1 M LiOD in D_2O 1.0 M LiOD in D_2O
2	Pd rod 1 mm dia, 7.5 cm long deformed in vice	Pt wire 0.8 mm dia 7 cm long	25	0.1 M LiOD in D_2O
3	Pd rod 1 mm dia, 7.5 cm long	Pt wire 0.8 mm dia 7 cm long	25	0.1 M LiOD in D_2O
4	Pd rod 1 mm dia, 7.5 cm long	Pt wire 0.8 mm dia 7 cm long	25	0.1 M LiOH in H_2O

2.1.3 Electrolyte Inventory Control/Other Equipment

Initially, a syringe/septum arrangement was used for make up of the volume of heavy water used up in the electrolysis. An automatic feed system was added late into the experimental program allowing continuous operation at ever increasing current densities over weekends. Off-gas deuterium and oxygen were removed from the cell through a tube dipped into a heavy water seal to reduce back diffusion of air containing oxygen and light water. It is possible that light water could have contaminated the cell through exchange with the heavy water seal so future work should consider a mineral oil seal used sucessfully by Huggins et al at Stanford. The electrolyte level was maintained near the top of the cell to minimize the amount of explosive gas mixture in the cell.

A data logging system was added for continuous measurement of the cell voltage, the cell current, the cell temperature given by a glass shielded thermocouple, and ambient temperature.

2.1.4 Radiation Measurements

A neutron bubble detector was initially installed in the cell but gave no useful results. A neutron detector supplied by Bicron was subsequently set up with a counter to monitor fast neutrons. The detector used an organic scintillant/photomultiplier combination technique to detect protons recoiling from collisions with incident neutrons. Gamma radiation was detected with a 7.5 cm x 7.5 cm sodium iodide crystal spectrometer and the gamma ray spectrum was recorded over three energy windows in the 0.1 to 4 Mev range using a Canberra series 30 analyzer. These windows were centred at 1.764 Mev to detect radiation produced by background Bi-214 decay, 2.614 Mev for gammas produced by Tl-208 decay and 2.2 Mev, the energy expected for the gamma from the n,H reaction. Any increase above the background level over 24 hours at 2.2 Mev was indicated by the ratio of the counts per minute at this energy to the total counts per minute at 1.764 and 2.614 Mev.

Tritium measurements were carried out by liquid scintillation counting of 1 mL samples in 20 mL of Hionic, a scintillant recommended for high ionic concentrations to avoid errors due to chemiluminescence.

2.1.5 Reference Electrodes

A palladium reference electrode was installed but interpretation of the results was uncertain because of its unknown hydride content. The cathode potential was also measured by a saturated calomel electrode which showed reliable operation but could possibly contaminate the electrolyte with chloride ions. Its use was therefore intermittent. Results from this electrode included an error because the Luggin capillary did not extend very closely to the surface of the Pd cathode which bent during the electrolysis.

2.2 Palladium Disc Electrode Experiments

The main objective of this series of experiments was to reproduce the results claimed by Huggins of Stanford University. The experimental set-up was very similar to that by Huggins with minor modifications. Two 1.9 cm ID and 5 cm long quartz tubes were used as the electrolysis cells. The two quartz cells were set up in a cooling bath with a palladium disc cathode in each cell, a platinum cage anode and lithium dissolved in light or heavy water as the electrolytes (see Figure 2). Temperatures of the two cells were monitored continously with thermocouples and recorded with a dual pen chart recorder.

2.2.1 Disc Cathodes

The cathode in each of the electrolysis cells was prepared by cutting a piece of about 2.3 grams of palladium from the same 10 oz bar used to prepare the rod electrode discussed above. The approximate size and shape of the four electrodes used are shown in Figure 3. Two electrodes (electrodes 1A and 2A)

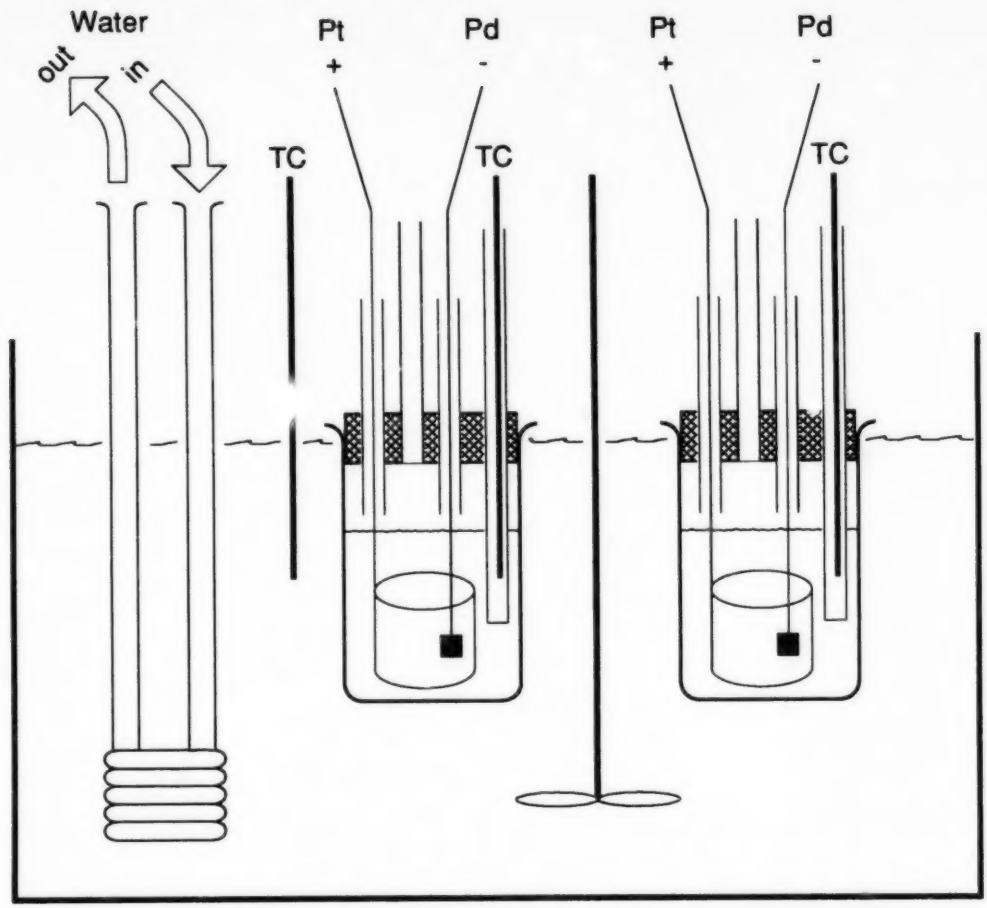

FIGURE 2

EXPERIMENTAL CALORIMETER ARRANGEMENT

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Electrode 1A

Approximate Thickness = 2 mm

Total Surface Area (Estimated) = 3.3 cm^2

Approximate Weight = 2.28 g

Electrode 2A

Approximate Thickness = 2 mm

Total Surface Area (Estimated) = 3.2 cm^2

Approximate Weight = 2.34 g

Electrode 1B

Approximate Thickness = 2 mm

Total Surface Area (Estimated) = 3.8 cm^2

Electrode 2B

Approximate Thickness = 2 mm

Total Surface Area (Estimated) = 3.5 cm^2

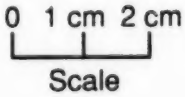

FIGURE 3

APPROXIMATE SIZE AND SHAPE OF THE DISC ELECTRODES

were prepared by the following procedure. Each piece of palladium was arc melted on a water cooled copper hearth in an argon atmosphere for 20 seconds for six times. Fine bubbles were observed on the surface of the side in contact with the copper hearth of the palladium. This surface was examined by Auger electron analysis. The smooth part of the surfaces was found to be mainly Pd whereas the bubbles contained oxygen or oxides. The samples were therefore polished with a 600 grit SiC abrasive paper. The surface of the polished palladium did not contain any oxygen or oxides but carbon particles were found to be embedded on the surface. A fine diamond paste (1 μm) was then used to further polish the surface. After the samples were polished, they were mechanically deformed by hammering to a disc shape of approximately 2mm thick. Two additional electrodes (electrodes 1B and 2B) were prepared following similar procedures except they were melted ten times instead of six for the first two electrodes and only diamond paste was used to polish the samples.

2.2.2 Electrolyte and Cell Operation

For the experiments with the first two disc electrodes 1A and 2A, cell 1A contained 0.1 M LiOH in H_2O and cell 2A 0.1 M LiOD in D_2O. For the experiments with the other two electrodes 1B and 2B, cell 1B was converted from H_2O with 0.1 M LiOH to D_2O with 1 M LiOD after 358.7 hours of operation. Small amounts of thiourea, iron sulfate and chromium sulfate were added to the cells after the cells had been operating for a period of time. A recombiner with Pd on alumina catalyst was installed in the off-gas line of one of the cells. The recombined D_2O was condensed and collected for tritium analysis. Details of the history of operation of the cells are summarized in Appendix C. Signficant operations during the experiments are summarized in Tables 2 & 3.

TABLE 2

SUMMARY OF THE OPERATION OF CELLS 1A AND 2A

TIME (Hour)	CELL 1A (0.1 M LiOH in H_2O)	CELL 2A (0.1 M LiOD in D_2O)	
0 to 403	Applied Voltage (V) = 3 to 8 V Cell Current (I) = 50 to 100 mA	V I = 70 to 145 mA	Charging the Pd electrodes at low current density of ~ 15 to 45 mA/cm^2
354.9	Cell Operation Temporarily Stopped	Cell Operation Temporarily Stopped	Electrolyte of both cells replaced with fresh stock solution
403 to 437	V = 8.5 to 11.2 V I = 650 to 1100 mA	V = 10.2 to 10.7 V I = 720 to 975 mA	Charging the Pd electrodes at higher current density of 200 to 300 mA/cm^2
437 to 545.4	V = 3 to 8.5 V I = 30 to 160 mA	V = 2 to 5.7 V I = 30 to 150 mA	Maintaining a low current density of 10 to 50 mA/cm^2
528	Cell Operation Temporarily Stopped	Cell Operation Temporarily Stopped	Electrolyte of both cells replaced with fresh stock solution
545.5 to 76.5	V = 7.78 to 7.84V I = 870 to 1040 mA	V = 9.0 to 9.3 V I = 740 to 860 mA	Increased the current density to 230 to 310 mA/cm^2
576.5 to 713.3	V = 3 to 3.8 V I = 67 to 180 mA	V = 3.5 to 4.5 V I = 60 to 160 mA	Maintaining a low current density of 10 to 50 mA/cm^2
671.3 to 689.9	Changed power supply at 671.3 h	Changed power supply at 689.9 h	Power supplies changed to achieve higher current density
713.3 - 737.8	V = 9 to 12 V I = 1.4 to 1.52 A	V = 11.7 to 13.5 V I = 1.22 to 1.4 mA	Increased the current density to 380 to 460 mA/cm^2
714.3	.01 g of Thiourea added	0.01 g of Thiourea added	Poisoning of electrodes attempted

TABLE 3

SUMMARY OF THE OPERATION OF CELLS 1B AND 2B

Time (hour)	Cell 1B (0.1 M Li OH in H_2)			Cell 2B (0.1 M Li OD in D_2O)			
	Applied Voltage(V)	Cell Current(A)		Applied Voltage(V)	Cell Current(A)		
0 to 48	3.9 to 4.4	0.23		4.3 to 5.1	0.21		Initial charging at low current densities of 60 mA/cm^2
48 to 100.8	12 to 15.8	2.45 to 2.55		12 to 19.4	2.13		Increased current density to 600 mA/cm^2
100.8 to 165.4	3.4	0.1		3.8	0.1		Maintained operation at a low current density
165.5 to 172.6	13 to 16.8	2.35 to 2.37	Polarity reversed at 170.9 h	16.7 to 18.8	2.13		Increased current density to 600 mA/cm^2
172.6 to 213.1	3.4 to 4.2	0.23		10 to 27.8	2.13	LiOD added to lower applied V	Cell 1B polarity reversed to desorb hydrogen Cell 2B maintain at high current density
213.1 to 219.3	13.9 to 14.1	2.3	Polarity reversed at 219.3 h	13.1 to 13.2	2.13	Thiourea added at 214. h	Cell 1B polarity reversed again to charge the Pd poison added to Cell 2B
219.3 to 245.9	13.9 to 15.9	2.3 to 2.6		12.2 to 17.5	2.13 to 2.3	LiOD added	Attempts were made to equalize the cell current and power to both cells
245.9 to 261.3	4	0.26		4.8	0.21		Current density lowered to 60 mA/cm^2
261.3 to 268.8	13.6 to 14.6	2.3 to 2.35		14.7 to 15.4	2.13 to 2.3	LiOD added	Current density increased
268.8 to 357.8	3.9	0.15	Polarity reversed at 269.0 h	3.6	0.15		Current density reduced, Cell 1B polarity reversed to desolve hydrogen
357.8 to 405.7	2.5 to 2.7	0.23	Converted to 1MLiOD at 357.8 h	3.9 to 4.3	0.21		Cell 1B converted to 1MLiOD in D_2O. Both cells at low current density
405.7 to 531.0	4.8 to 6	2.3	Thiourea added at 507.1 & 531 h	3.9 to 4.5	0.21		Cell 1B at high current density with thiourea added twice Cell 2B maintained at low current density started using meter pump to feed D_2O to both cells
531 to 532.7	6.1	2.3		16.1	2.1		Cell 2B current density increased
532.7 to 549.2	6.4	2.3		4.9	0.21		Cell 2B current density decreased temporarily

TABLE 3

SUMMARY OF THE OPERATION OF CELLS 1B AND 2B (Cont'd)

Time (hour)	Cell 1B (0.1 M Li OH in H_2)			Cell 2B (0.1 M Li OD in D_2O)			
	Applied Voltage(V)	Cell Current(A)		Applied Voltage(V)	Cell Current(A)		
549.2 to 603.6	5.9 to 6.5	2.3	$FeSO_4$ in solution added at 577.5 h lost electrolyte at 596 to 597 h	18.3 to 30.1	2.1	$FeSO_4$ in solution added 577.5 h	Fe added to both cells in an attempt to form dendrites
603.6 to 66.94	2.5 to 2.7	0.23		7.4 to 9.6	0.21		Current densities in both cells reduced
669.4 to 772.7	6.4 to 8.2	2.3	Thiourea added at 669.4 and 765.5 h $Cr_2(SO_4)_3$ in solution added at 698.5 h Thiourea	13.5 to 30	1.4 to 2.1	Thiourea added at 669.4 and 765.5 h LiOD added at 669.5, 672.4, 675.7, 742 h $Cr_2(SO_4)_3$ in solution added at 698.5 h	Current densities were increased with addition of Thiourea (twice) and $Cr_2(SO_4)_3$ in solution

3.0 RESULTS AND DISCUSSION

3.1 Palladium Rod Electrode Experiments

Detailed results for cells 1 to 4 are given in Appendix A and B. The **heat balance** calibrations showed that no excess heat was being generated within 0.25 watts in 18 watts or 1%.

In an effort to increase the deuterium content of the cell 1 cathode, (Pd rod, 0.6 cm dia.) various treatments were tried. These included:

(a) reversing the current recommended in some texts to remove impurities,

(b) palladizing the cathode by depositing a thin layer of Pd black,

(c) pounding some of the cathodes as was carried out by Stanford,

(d) adding other cations to grow dendrites on the electrode surface,

(e) adding small amounts of arsenic and thiourea poison which inhibit the recombination to D_2 of D atoms at the electrode surface,

(f) increasing the molarity of the lithium deuteroxide in heavy water to one molar to allow large increases in current density, and

(g) cycling the cell current between low and high values.

Weight increase after four weeks of electrolysis showed an overall D/Pd ratio of 0.64. This figure may be high due to the presence of impurities plated on the surface of the Pd cathode. The importance of using suitable glass for the cells was well demonstrated by the calcium silicate deposits found on the cathodes of cells 2, 3 and 4. Following this finding, tests with these cells were discontinued. As is well known, the electrode was bent due to the deuterium ingress. At the end of the eleven week test period, the deuterium to palladium atomic ratio had risen to 0.78 determined by collecting the deuterium desorbed on heating the electrode.

The calomel electrode showed that the surface of the Pd cathode was exhibiting a constant potential of -0.8 volts which could be lower in reality after correction for the distance between the electrode surface and the point of measurement of the Luggin capillary in the electrolyte. Fleishmann and Pons report that -2.0 volts may be possible.

In addition to the observation of no excess heat, the **neutron count** rate was not significantly affected although the background tended to increase from about 5.0 to 5.6 counts/min over the first month of the test and fluctuate somewhat depending on counting time.

The **gamma counts** in the 2.2 Mev range did not change significantly from the value of about 0.29 of the total counts in the adjacent ranges which was the value before the electrolysis was initiated indicating no detectable contribution of an n,H reaction.

Similarly, the **tritium activity** in the electrolyte increased to only about three times the background level in the make up heavy water which is similar to the expected level of tritium in a separation process based on electrolysis and much lower than the several orders of magnitude increase reported by Texas A&M.

3.2 Palladium Disc Electrode Experiments

The main purpose of these experiments was to reproduce the results claimed by Huggins at Stanford. Equipment used and procedures followed in these experiments were based on the information available on the experiments carried out by Stanford. Initially, the heat effect in the D_2O cell was compared with the H_2O cell at conditions reported by Huggins. Two sets of electrodes in two sets of cells (electrodes 1A and 2A in cells 1A and 2A, respectively, and electrodes 1B & 2B in cells 1B and 2B, respectively) were tested. These electrodes were 2 mm thick discs of about three grams in weight. Cell 1A and 1B contain 0.1M LiOH in H_2O while cell 2A and B contained 0.1 M of LiOD in D_2O. The electrolyte in cell 1B was later changed from 0.1M LiOH in H_2O to 1M LiOD in D_2O to study the effect of concentration of LiOD on the performance of the cell. Significant operations of the cells are summarized in Tables 2 and 3.

3.2.1 Temperature Comparisons with Disc Electrodes

The temperatures of the two cells were monitored and compared continuously. For the first set of cells, cell 1A and 2A, at the low current density of 50 mA/cm^2 or less, the difference between the temperatures of the two cells was less than 0.2°C. When the current densities were increased to 200 to 460 mA/cm^2, with the power to each cell equalized, the temperatures in the two cells were found to be identical in the beginning. However, the temperature in cell 1A tended to increase at a slightly faster rate. This was because the current was slightly higher in this cell in order to maintain the same power input to the two cells. The higher current led to a higher electrolysis rate in this cell and caused the electrolyte level to drop at a slightly faster rate. The lower electrolyte level led to a slightly higher temperature because the same power input was heating less electrolyte and the heat loss from the cell to the water bath was also less. The temperature in the H_2O cell (cell 1A) was as much as 2°C higher than that in the D_2O cell (cell 2A) after operating at the high current density for about 10 hours.

For the second set of cells, cell 1B and 2B, at low current density of 60 mA/cm^2, the temperature difference was again less than 0.2°C. However, the temperature difference at high current density of about 600 mA/cm^2 and same power input seemed to fluctuate and the temperature in cell 1B with 0.1 M LiOH was sometimes higher and sometimes lower than the temperature in cell 2B. Several factors seemed to affect the temperatures in the cells at the same power input to the cells such as the amount of electrolyte in the cell and the cell current which affected the rate of gas generation, hence, the mixing and heat transfer of the cell. The location of the thermcouple in the cell might also affect the temperature readings depending on whether the cell was well mixed or not. Furthermore, at the high current density of 600 mA/cm^2, the electrolysis rate was very high and it was necessary to add H_2O and D_2O every hour to the two cells 1B and 2B, respectively. This also affected the temperatures in the two cells. An attempt was made to equalize both the power and current of both cells by adding LiOD to cell 2B which had a slightly higher voltage drop. The electrolyte levels in the two cells were also controlled to be approximately the same. After these adjustments were made, the temperature in the two cells differed by less than 1°C. But again the temperatures in the two cells were fluctuating such that sometime cell 1B was at a higher temperature and sometimes cell 2B was higher.

3.2.2 Tritium Measurements with Disc Electrodes

Based on the above results, it was concluded that because of the many factors affecting the temperature in the cell, the present calorimetry set-up could not provide sufficiently accurate results to indicate whether excess heat was produced in one cell over the other. It was therefore decided to convert cell 1B to 1M LiOD in D_2O and concentrate on tritium measurements of the recombined vent gases and of the electrolytes in the two cells. Thiourea, $FeSO_4$ and $Cr_2(SO4)_3$ in solution were also added to the cells at different times to poison the electrode and in an attempt to grow dendrites on the electrode that might possibly lead to some surface effect. Table 4 summarizes the tritium concentrations of the various samples taken at different times. Results showed that the tritium concentration in the D_2O used to prepare the electrolyte

was about 150 nCi/L. The recombined D_2O from the cells generally had a lower tritium concentration as low as 48 nCi/L. This is expected as the electrolysis process tends to selectively electrolyse light water. Two samples of the electrolyte had a tritium concentration of 170 nCi/L while one sample was 128 nCi/L. None of the samples showed any significant increase of tritium concentration.

TABLE 4

TRITIUM CONCENTRATIONS IN THE VARIOUS LIQUID SAMPLES

Sample Identification	Date	Time (h)	Run Time (h)	Tritium Concentration nCi/L
Recombined D_2O from Cell 2B	Aug 24	16:00	77.6	120
Recombined D_2O from Cell 2B	Aug 24	21:30	83.1	100
Recombined D_2O from Cell 2B	Aug 25	7:45	93.4	120
D_2O Blank	Aug 25	-	-	150
Recombined D_2O from Cell 2B	Aug 25	8:25	94.4	150
Cell 2B Electrolyte	Aug 29	8:30	190.1	170
0.1 M LiOD stock solution	Aug 29	-	-	130
Recombined D_2O from Cell 2B	Aug 28	21:40	179.4	150
Recombined D_2O from Cell 2B	Aug 29	7:45	189.4	140
Recombined D_2O from Cell 2B	Aug 28	14:00	171.6	130
Recombined D_2O from Cell 2B	Sept 7	15:30	413.1	48
Recombined D_2O from Cell 2B	Sept 8	9:00	430.6	58
Recombined D_2O from Cell 2B	Sept 9	11:05	456.7	73
Recombined D_2O from Cell 2B	Sept 10	13:00	482.6	102
Recombined D_2O from Cell 2B	Sept 11	7:40	501.3	82
Recombined D_2O from Cell 1B	Sept 12	7:50	525.4	91
Recombined D_2O from Cell 1B	Sept 13	7:40	549.3	97
Recombined D_2O from Cell 2B	Sept 13	9:50	551.5	100
Recombined D_2O from Cell 2B	Sept 14	7:43	573.3	100
Recombined D_2O from Cell 2B	Sept 15	7:40	597.3	109
Recombined D_2O from Cell 2B	Sept 18	7:34	669.2	95
Recombined D_2O from Cell 1B	Sept 19	7:24	693.0	91
Recombined D_2O from Cell 2B	Sept 19	12:55	698.5	76
Recombined D_2O from Cell 2B	Sept 20	7:45	717.4	106
Recombined D_2O from Cell 1B	Sept 21	8:05	741.7	97
Recombined D_2O from Cell 2B	Sept 22	7:50	765.5	91
Recombined D_2O from Cell 1B	Sept 22	15:06	772.7	96
Cell 1B electrolyte	Sept 22	-	772.7	170
Cell 2B electrolyte	Sept 22	-	772.7	128

4.0 CONCLUSIONS

No excess heat nor tritium production has been found to date but good progress has been made up the learning curve with respect to acceptable heat measuring techniques, radiation measurements and diagnostics for deuterium content of the palladium and how to increase it.

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APPENDIX A

SUMMARY OF RESULTS FOR CELL 1

Day	Month			Neutron Count Count/Min
	Apr	Tues 25	Pd rod heat treated 800°C/18 hrs/low vacuum; cleaned in acetone (rod is 0.6 cm dia x 7 cm long, wt 21.7 g, vol 1.98 cm^3). Background neutron count in adjacent building =4.90 counts/min.	
1		Fri 25	Electrolysis started using electrolyte prepared from Li metal removed from oil, wiped clean, reacted with D_2O and top layer discarded; initial difficulty getting current; reason unknown, may be due to low set of temperature switch; suddenly gassing seen at cell -unsure whether anode or cathode; I ~ 23 mA/cm^2; 25 minutes after electrolysis started Pd cathode vs Pd ref = -1.17 V five seconds after disconnecting power;	
4	May	Mon 1	Cell Temperature = 32°C.	4.95 over 72 hours
6		Wed 3	Carried out Pd cathode vs Pd ref check for ~ 15 mins; power disconnected: voltage constant for ~ 1 minute at -1.18 V then decreased gradually to -0.4 V then suddenly plunged to zero at which significant gassing seen at cathode!	
7		Thurs 4	Cells 2, 3, 4 started; 25 mL glass sample bottles of soft glass; all 1 mm dia Pd wire, 7.5 cm long bent into U shape; all cathodes heat treated in vacuum at 1000°C for 2 hours; cells 2,3 are 0.1M LiOD in D_2O ; cell 4 is H_2O; cell 2 Pd cold worked by mechanical squeezing in vice, other cells Pd as is; no heater in cells 2,3,4. Neutrons = 4.94 counts/min on cell 1 averaged over 24 h.	4.94 over 24 hours
8		Fri 5	Arsenic added to cell 1 electrolyte: 3 mL of 0.03 molar; no great effect seen; delta V across cell dropped ~ 0.05 Volts;	

	11	Mon 8	Polarity of all four cells changed 10 times (10 mins at each polarity); it required about 4.2 V to drive 0.32 amps with Pd as the cathode but only 2.7 V to drive 0.32 amps with Pd as the anode. Particles appear in cells during this procedure. (Black PdO?).	5.06
	12	Tues 9	Increase cell 1 current density to ~ 70 mA/cm^2. T= 47°C in room air.	5.08
	15	Fri 12	Measured gas rate agreed with electrolysis current; Bubbles appear smaller for solution is cloudy but clears with current off. Added 1 mL Pd $(NO_3)_2$ ~ 0.003 g/mL to palladize cell 1 cathode; it appeared that the anode Pt was discoloured rather than cathode: why?	5.27
	18	Mon 15	Cell 1 heater on; carried out heat balance → no excess heat seen.	5.27
	19	Tues 16		5.18
19	21	Thurs 18	Saturated Calomel Electrode installed; shows D present in electrode → -1.13 (SCE); cf -0.051 V obtained with Pd rod (must add 0.242 V to convert to NHE) gives -0.89 V.	5.14
	26	Tues 23	$V_{cathode}$ (NHE) = -0.84 V (NB as cell conductivity goes up ohmic loss falls)	
	27	Wed 24	$V_{cathode}$ (NHE) = -0.86 V	
	28	Thurs 25	Weighed cell 1 electrode → Pd/D ~ 0.64; <u>NB electrode was bent</u>. X ray fluorescence → Pb, Pt, Zn, Cu present on surface (due to solder). Clean electrode surface with emery paper and replace in electrolyte; gave bubbles as H_2 is lost with current off; $V_{cathode}$ (NHE) = -0.68; restart electrolysis.	
	29	Fri 26		5.65
	32	Mon 29		5.58 over 72 hours
90-28	33	Tues 30	Heat balance on cells 2, 3, with cell 4 as reference showed no excess heat.	5.74

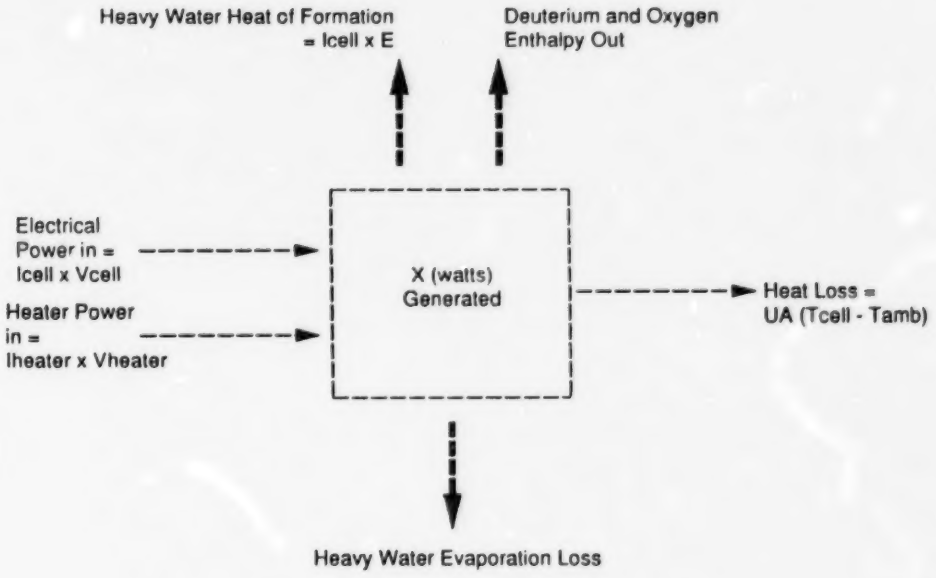
34		Wed 31	Start forced cooling on cell 1 with fan, T= 39°C; cell 3 stopped; Pd had calcium silicate on surface due to glass bottle; some Zn found also (may be solder); scale flaked off easily; bare metal Pd had sulphur! (ICP showed Zn & Ca in the Lithium added to the D_2O).	5.72
35	June	Thurs 1	High amp power supply added to cell 1! I ~ 300mA/cm2. Cell 2 & 4 discontinued. Timed electrolyte addition started to cell 1. ICP showed no Zn, Cu, Ca and Si in D_2O but 0.2 ppm Ca and .02 ppm Zn in 0.1 M LiOD in D_2O.	5.82
35-39		Fri - Mon 5	Cell 1 temp ~ 65°C; fine bubbles keep cell milky;	5.83
39		Mon 5	Add water jacket around cell; temp reduced to ~ 35°C. Electrolyte conductivity ~ 8.7 mS/cm dropped from 11.7 mS/cm; add 5 mL of 1 M LiOD to increase conductivity to 11.4 mS/cm. Copious large bubbles from Pd with power off.	
40		Tues 6	Cell 1 has lost its milky appearance; looks clear due to large bubbles! With power off, fine bubbles given off at low rate. May have been caused by the lower temperature or organics in the 1 M LiOD!	5.72
41		Wed 7	Electrolyte conductivity 12.27 mS/cm;	5.71
42		Thurs 8	Remove electrode and weigh → no further increase in weight in 2 weeks; sand to bright surface, reduce length to 5.5 cm; remove 0.1 M electrolyte and replace with 1 M LiOD in D_2O; current density 357 mA/cm^2; no increase in T2 content of 0.1 M LiOD electrolyte. ICP analysis of 0.1 M electrolyte 3 ppm Sn, 22 ppm Na, 0.3 ppm Pt, 9 ppm Al.	
43-46		Fri - Mon 12	Calomel voltage seems to be increasing or strongly temperature dependent.	
47		Tues 13	Pd electrode pounded and surface deformed with flat spots, out of round; it was still gassing when replaced in cell with current off; also SCE reading was -0.906 cf -1.25 just before Pd electrode removal for pounding;	
48 53		Wed 14 Mon 19	Electrolysis continued with auto electrolyte make-up (tritium content of make-up D_2O is ~ 120 dpm/mL D_2O).	
53 54		Mon 19 Tues 20	Heat balance → no excess heat ± 0.25 watts.	

55		Wed 21	0.2 g $Cu(NO_3)_2.1/2\ H_2O$ added to cell → immediate green precipitate which turned reddish brown in 90 minutes; continue electrolysis;	5.61
56		Thurs 22		5.61
61		Tues 27	Increase current density to 750 mA/cm^2; using up a lot of D_2O.	
63		Thurs 29	Data logger problems; repair anode lead; D_2O make up tritium content is 170 dpm/mL; clean black particles from cell bottom.	
64		Fri 29	Ran at 320 mA/cm^2 over long weekend.	5.80
68	July	Tues 4		
68 70		Tues 4 - Thurs 6	Cycle current density 320 to 90 to 750 to 90 to 750 mA/cm^2; no noticeable effect.	
70		Thurs 6	Sand cathode free of black deposit; add 0.05 g thiourea.	
71		Fri 7	Add another dose of thiourea; reduce current to 320 mA/cm^2 over weekend; add another dose of thiourea; thiourea increases cell resistance.	
74		Mon 10	Increase current density to 750 mA/cm^2; decrease current to 90 mA/cm^2 overnite.	
74		Tues 11	Increase current to 750 mA/cm^2. decrease current density to 90 mA/cm^2 overnight.	5.8 overnight 5.6
76		Wed 12	Current increased to 750 mA/cm^2; electrolyte tritium ~ 515dpm/mL; added 0.05 g thiourea.	5.6 overnight
77		Thurs 13	Electrolyte tritium = 485 dpm/mL.	5.8 (24 h) 5.91 (5 h) 5.57 (2 h)
78		Fri 14	Heat balance showed no excess heat; give up; remove cathode; heat to redness; → D/Pd = 0.78; ~1650 mL D_2(NTP); after cell switched off then 5.6 counts/min averaged over the next 1.25h.	5.8 (16 h) 5.6 (1 h) 5.8 (2 h) 5.3 (2.5 h) 5.6 (1.25 h)

APPENDIX B

HEAT BALANCE CALCULATIONS

The schematic below shows the heat balance over the electrolytic cell assuming an internal rate of heat generation of X, a constant heat transfer coefficient U (watts/cm^2/°C) for heat losses and a potential (E=1.54 volts) for electrolysis of heavy water.



Equating the total power in with the energy out gives:

$$Q_{heater} + I_{cell}(V-E) - (D_2O \text{ Evaporation Loss}) - D_2/O_2 \text{ Enthalpy out} = UA(T_{cell}-T_{amb}) - X$$

This is an equation of a straight line of slope UA and intercept equal to -X.

Typical calibration data are given in Table B.1. These data are plotted in Figure B.1. which shows no excess heat generation within 0.2 watts.
Based on the claims of Fleischmann and Pons and Huggins, a ten percent excess energy should have resulted in an intercept at -1.7 watts.

TABLE B1

TYPICAL EXCESS HEAT CALIBRATION DATA

	Heater Off	Heater On
CELL CURRENT (amps)	3.98	3.931
CELL VOLTAGE (volts)	6.037	5.79
ELECTROLYSIS POWER (watts)	17.90	16.71
HEATER POWER (watts)	0	11.68
CELL TEMP (deg C)	33.45	39.02
AMB TEMP (deg C)24.3524.66		
Tcell - Tamb (deg C)	9.1	14.36
D2 PRODUCED (gms/sec)	0.00002	0.0002
O2 PRODUCED (gms/sec)	0.000082	0.000081
SENSIBLE HEAT LOSS (watts)	0.002026	0.00332
EVAPORATION LOSS (moles D2 + 02/sec)	7.0E-06	7.0E-06
D2O EVAPORATED (moles/sec)	1.0E-06	1.0E-06
EVAP HEAT LOSS (watts)	0.044392	0.043846
HEATER + ELECTROLYSIS POWER - SENSIBLE HEAT - EVAP HEAT LOSS (watts)	17.85	28.34

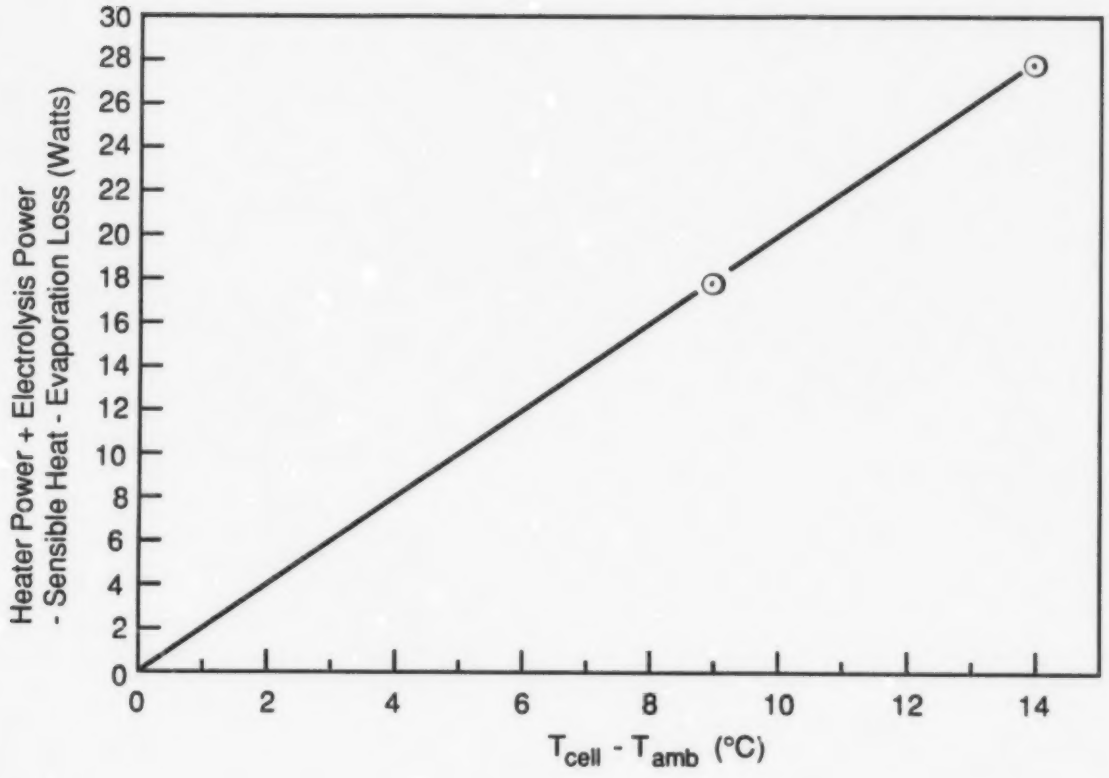


FIGURE B-1

PLOT OF ACCOUNTABLE ENERGY VS CELL TEMPERATURE RISE

APPENDIX C

SUMMARY OF DISC ELECTRODE EXPERIMENTS

Tables C1 and C2 summarize the operation history of cells 1A and 2A and cells 1B and 2B, respectively. The cell current and voltage are plotted in Figures C1 and C2, for cell 1A, in Figures C3 and C4 for cell 2A, in Figures C5 and C6 for cell 1B, and in Figures C7 and C8 for cell 2B.

Table C1. Detailed Operation History of Cells 1A and 2A

Month	Date	Hour	Run time	Cell#1 Voltage (V)	Current (mA)	H2O (mL)	Cell#2 Voltage (V)	Current (mA)	D2O (mL)	Comments
July	18	14.3	0.0	3	149.8		3.1	150.2		
	19	7.9	17.7	3	107.6		3.1	130		
	19	8.0	17.8	3.1	142		3.3	143.1		
	19	11.8	21.6	3	149.2		3.4	140.9		
	19	15.3	25.0	3	140.4		3.4	143		
	20	7.7	41.4	3	144.3		3.4	138.5		
	20	8.0	41.8		142.5	2		140.3	2	
	20	8.1	41.9		141.9			140.8		
	20	11.3	45.1	3	137.6		3.5	142.3		Tbath=23C, Cell1&2=23C
	20	15.1	48.8	3	139.6		3.5	140		
	21	7.7	65.4	3	138.1		3.5	134		Tbath=23C, Cell1&2=24C
	21	13.5	71.3	2.8	74.9	5	3	70.5	4	
	24	7.7	137.5	2.7	54.2	4	2.9	69.4	1.5	
	24	12.2	142.0	3.5	141.2		3.8	142.4		
	24	12.4	142.1	3.5	141.9		3.8	138.1		
	24	15.2	145.0	3.5	140.4		3.8	140.8		
	25	7.8	161.5	3.4	129	3	3.7	134.1	3	
	25	8.1	161.9		142.6			140.6		
	25	11.9	165.7	3.6	148.6		3.8	140.7		
	25	15.1	168.8	3.5	141.3		3.8	139.8		
	26	7.6	185.4	3.5	124.4	3	3.8	121.8	2.2	
				3.7	141.8		4.1	141.3		
	26	13.1	190.8	3.7	142.9		3.8	152		
	26	15.1	192.9	3.7	143		3.9	141.2		
	27	7.6	209.3	3.7	139.9	3	3.9	137.2	2	
				3.9	140.5		4.2	142.2		
	27	11.9	213.7	3.8	141.4		4.2	141		
	27	15.0	216.8	3.8	140.3		4.1	141.5		
	28	7.6	233.3	3.8	135.3	4	4	139.6	2.8	
	28	7.8	233.5	4.2	140.4		4.4	141.2		
	28	9.8	235.5	4.309	141.5		4.489	139.5		
	28	13.1	238.9	4.307	140.2		4.556	140		
	28	15.0	240.8		139.1		4.549	147		Tbath=24C, Cell#1&2=25C
	28	15.3	241.0	3.482	71.6	3	3.567	71.3	2	
	31	7.8	305.6	3.4	50.2	7	3.562	71.6	2.3	
	31	8.1	305.8	6.367	140.5		4.739	140.3		
	31	12.0	309.7	6.25	143		4.73	145.2		
	31	14.8	312.5		136.4					Tbath=23/24C, Cell#1&2=25/26C
	31	14.9	312.6	6.341	139					
	31	15.2	313.0				4.729	144.5		
	31	15.3	313.0	6.884	139	2		143.7		
August	1	7.6	329.4	6.869	142	2.7	4.729	123.1	5	
	1	8.2	330.0	7.11	141.4		5.674	142		
	1	9.8	331.6							
	1	9.9	331.6				5.558	140.9		Collecting gas from cell#2
	1	10.9	332.7							96 mL in 59 min
	1	11.3	333.1		140.8					Collecting gas from cell#1
	1	12.3	334.1							87 mL in 60 min
	1	11.8	333.6							bath=327uS/cm
	1	14.9	336.6	7.131	139.2	1.5	5.553	140.6	2.3	
	1	15.1	336.9	7.777	140.9		5.86	141.6		
	1	15.5	337.2							
	2	7.9	353.7	7.765	140.1	3				
	2	8.0	353.8	8.184	141.6		5.856	123.1	4.5	
	2	8.3	354.0				7.099	141.5		
	2	9.0	354.7							C#1 off, teflon removed
	2	9.2	354.9							C#2 off, teflon removed
	2	11.1	356.8	2.914	142.3					fresh electrolyte-both cells
	2	11.3	357.0				3.422	141.4		

Table C1. Detailed Operation History of Cells 1A and 2A (Cont'd)

Month	Date	Hour	Run time	Cell#1 Voltage (V)	Current (mA)	H2O (mL)	Cell#2 Voltage (V)	Current (mA)	D2O (mL)	Comments
	2	15.5	361.3	3.087	139		3.596	141.8		
	3	7.7	377.4		119.2		3.586	126.9		
	3	7.8	377.5	3.23	140.4	1.8			1.4	
	3	7.9	377.6				3.789	143.2		
	3	12.9	382.7	3.177	99.6		3.78	112.2		
	3	13.1	382.8	3.464	142		4.073	141.5		
	3	13.6	383.4	3.462	140.9	1.9	4.031	141.2	1.8	
	4	7.7	401.5	3.472	136.3		4.042	142.2		
	4	8.1	401.8							stirrer in bath started
	4	8.1	401.9	3.657	141.8					
	4	9.3	403.0	8.635	1016		10.19	844		
	4	15.4	409.2	8.581	1004	4.9				
	4	15.6	409.3				10.13	884	3.5	
	4	16.3	410.0	9.513	974		10.701	841		
	4	22.2	415.9	9.489	1144	6.5	10.66	975	3.5	
	4	22.5	416.3	8.897	802		10.66	804		Tbath=18C
	4	22.8	416.5	9.78	848		10.667	805		
	5	8.4	426.2	9.752	935	10.5	10.656	889	5	
	5	9.2	426.9	11.193	650		10.661	719		
	5	19.4	437.2	11.177	738	6	10.652	845	3.5	
				3.236	36.8		3.059	41.3		
	7	8.3	474.1	3.256	40	6	3.08	36.2	5	
	8	7.8	497.6	3.254	28.1	2.5	2.077	32.5	2	
				7.398	142.6		5.309	141.8		
	8	11.8	501.5	7.384	134.1		5.295	124.3		
	8	15.3	505.1	7.6	147.8		5.669	143.6		
	9	7.7	521.4	7.593	162.9	2.5	5.665	154.8	2.6	
	9	8.0	521.8		143.9		5.746	138.9		
	9	11.3	525.1							C1 off for modifications
	9	13.0	526.8	8.511	142.5					C1 on
	9	13.9	527.6							C2 off for modifications
	9	14.2	527.9					132.5		C2 on
	9	14.5	528.2							C1&2 off, change electrolyte
	9	14.6	528.4		140					C1 on
	9	14.7	528.5					141		C2 on
	9	15.1	528.9	3.374	140.8					
	9	15.2	529.0				3.833	140.3		
	10	7.6	545.4	3.364	141.1	1	3.82	120.7	1.1	
	10	7.8	545.6		142.5		10.338	1007		
	10	7.9	545.7	7.841	1002					
	10	8.1	545.8				9.205	806		
	10	8.4	546.2	7.831	977					
	10	8.6	546.4				9.346	797		
	10	11.3	549.1	7.813	1017		9.333	857		
	10	11.5	549.2				9.162	833		
	10	13.1	550.9	7.805	1024		9.155	830		
	10	14.9	552.6	7.8	1041					
	10	15.0	552.7			2.6				
	10	15.0	552.7				9.151	842	2.1	
	10	15.5	553.2	7.8	873	2.5				
	10	15.7	553.5				9.147	738	2	
	11	7.5	569.3	7.778	1028					
	11	7.6	569.4			3.9				
	11	7.7	569.5				9.128	892	3.1	
	11	7.8	569.6							
	11	8.3	570.0	7.778	925		9.128	785		
	11	8.3	570.1				8.994	767		
	11	11.7	573.5	7.778						replace fuse in C1
	11	11.8	573.6	7.782	967		8.982	796		

Table C1. Detailed Operation History of Cells 1A and 2A (Cont'd)

Month	Date	Hour	Run time	Cell#1 Voltage (V)	Current (mA)	H2O (mL)	Cell#2 Voltage (V)	Current (mA)	D2O (mL)	Comments
	11	12.1	573.8				9.092	812		
	11	14.7	576.5	7.778	955	2.6				
	11	14.8	576.6	3.606	142.2					
	11	14.9	576.7				9.084	818	2.1	
	11	15.1	576.8				4.14	140.2		
	11	15.3	577.1	3.062	67.4					
	11	15.5	577.2				3.487	72.4		
	14	7.5	641.3	3.065	83.2	2.2	3.494	61.2		
	14	7.8	641.6	3.599	142.5		4.366	141.8	1.7	
	14	14.1	647.9	3.593	121.1		4.358	135.6		thermocouple tube changed
	14	15.0	648.8	3.746	140.3		4.423	141.8		
	15	7.5	665.3	3.744	132.9	1.4	4.418	160.4		
	15	7.8	665.5	3.836	142.5		4.305	142.3	1.4	
	15	13.5	671.3	3.832	183.4		4.295	149.4		new power supply tested in C1
	15	14.9	672.7	3.554	140.9		4.223	141.4		
	16	7.7	689.4	3.542	166.3	1.4				
	16	7.8	689.6				4.216	137.8	1.3	
	16	8.2	689.9							C1&2 power supply:B&K 1635
	16	8.4	690.1				3.8	140		
	16	8.4	690.2	3.2	140					
	16	15.3	697.0	3.2	140		4	140		
	17	7.5	713.3	3.2	140	1	3.9	140	1.4	
	17	7.6	713.3	9.7	1400		13.1	1400		
	17	7.8	713.5	9	1400		11.8	1400		
	17	7.9	713.7	9.5	1520		11	1270		
	17	8.6	714.3							ThioU:.2mL(1g/20mL) to C1&2
	17	9.7	715.5	11.2	1520		13.1	1270		
	17	11.3	717.1							Tbath=26C
	17	12.0	717.8	11.4	1520		13.2	1270		Tbath=18C
	17	13.3	719.1							Tbath=16C
	17	13.5	719.2	11.1	1520		13.2	1270		T1>T2
	17	13.9	719.7							thermocouple interchanged
	17	14.2	719.9	11.1	1500		13	1270		
	17	14.4	720.2							thermocouple interchanged
	17	14.5	720.3							
	17	14.6	720.4			3.1				
	17	14.7	720.5						2.4	
	17	14.8	720.6	11.7	1410					
	17	15.1	720.8				13.4	1220		
	17	20.7	726.4	10.5	1410	5	12.2	1220	4	
	17	20.8	726.6	12.1	1410		13.5	1220		
	18	7.5	737.3	10.3	1410		11.7	1220		Tbath=18C
	18	7.7	737.5	11.4	1410	4.4	12.8	1220	3.4	
	18	8.1	737.8							Electrodes in C1&2 changed

Table C2. Detailed Operaton History of Cells 1B and 2B

Month	Date	Hour	Run time	Cell#1 Voltage (V)	Current (A)	H2O (mL)	Cell#2 Voltage (V)	Current (A)	D2O (mL)	Comments
August	21	10.4	0.0		0.23			0.21		Tbath=20C
	21	11.1	0.7	3.9	0.23		4.3	0.21		
	21	13.7	3.3	4	0.23		4.3	0.21		
	21	15.2	4.8	4	0.23		4.4	0.21		Tbath=20C
	22	7.5	21.1	4.1	0.23	1.6	4.7	0.21	1.2	
	22	7.9	21.5	4.1	0.23		4.8	0.21		
	22	8.0	21.6						0.3	C2 disconnect momentarily
	22	8.6	22.2	4.1	0.23		4.9	0.21		
	22	12.8	26.4	4.1	0.23		4.9	0.21		Tbath=19C
	23	7.5	45.1	4.3	0.23		4.8	0.21		
	23	7.7	45.3	4.412	0.23	1.9	5.102	0.21	1.8	
	23	8.5	46.1	4.418	0.23		5.072	0.21		
	23	10.2	47.8	4.408	0.23		5.038	0.21		Tbath=18.5C
	23	10.5	48.1		2.45			2.1		
	23	11.1	48.7	14.76	2.45		16.68	2.1		Tbath=20.5C
	23	11.5	49.1	15	2.45	0.8	16.8	2.1	0.7	
	23	13.7	51.3	14.2	2.45	1.7	14.2	2.1	1.6	Tbath=24C
	23	14.0	51.6	14.97	2.45		16.65	2.14		Tbath=25C
	23	14.7	52.3							water flow in bath increased
	23	14.8	52.4							Tbath=18C
	23	14.9	52.5	15.4	2.45		17.4	2.13		
	23	15.0	52.7	15	2.45		17	2.13		water flow in bath reduced
	23	15.7	53.3			5.5			5	
	23	15.9	53.5	17	2.45		19.4	2.13		Tbath=20C
	23	21.3	58.9	13.9	2.45	5	16.4	2.13	4.5	Tbath=27C
	23	22.1	59.8	15.75	2.55		18.5	2.13		
	24	7.5	69.1	13.4	2.55		14.8	2.13		Tbath=29C
	24	7.5	69.1			7			4.3	
	24	8.0	69.6							
	24	8.2	69.8	15.3	2.55		17.4	2.13		Tbath=25.5C
	24	8.8	70.4			1			0.8	Tbath=23C
	24	9.8	71.4	15.6	2.55		18	2.13		Tbath=22C
	24	9.8	71.4			0.8			0.7	
	24	10.4	72.0	15.7	2.55		18.2	2.13		
	24	10.9	72.5		2.55	0.8		2.13	0.7	Tbath=19C
	24	11.8	73.4	15.8	2.55		18.4	2.13		Tbath=17C
	24	11.8	73.4			0.8			0.7	
	24	12.5	74.1							stopped water flow
	24	13.4	75.0	14.6	2.55		17.2	2.13		Tbath=24C
	24	14.1	75.7			1.8			1.6	Tbath=27C
	24	15.0	76.6	14.2	2.55		16.4	2.13		Tbath=30C
	24	15.1	76.7			0.8			0.8	
	24	15.4	77.0	14.2	2.55		16.4	2.13		Tbath=32C
	24	16.0	77.6			5			5	
	24	16.3	77.9	15.25	2.55		18.1	2.13		
	24	16.5	78.1	15.1	2.55		17.75	2.13		Tbath=36C
	24	21.5	83.1	12	2.55	7.5	14.1	2.13	5.5	Tbath=48C
	24	22.2	83.8	14.75	2.55		16.55	2.13		
	24	22.2	83.8	14.75	2.55		16.8	2.13		
	25	7.5	93.1	12.1	2.55		12.2	2.13		Tbath=53C
	25	7.8	93.4			6.6			4.2	
	25	8.0	93.6	13	2.55		14.5	2.13		Tbath=49C
	25	8.8	94.4	12.6	2.5		14.2	2.13		
	25	8.8	94.4		2.45					Tbath=46C, water flow started
	25	9.0	94.6			0.8			0.7	Tbath=46C
	25	9.7	95.3	13.3	2.45		15.2	2.13		Tbath=41C
	25	10.1	95.7			0.8			0.7	
	25	10.7	96.3	13.5	2.45		15.5	2.13		Tbath=38C
	25	11.1	96.7			0.8			0.7	

Table C2. Detailed Operaton History of Cells 1B and 2B (Cont'd)

Month	Date	Hour	Run time	Cell#1 Voltage (V)	Current (A)	H2O (mL)	Cell#2 Voltage (V)	Current (A)	D2O (mL)	Comments
	25	12.1	97.7	13.9	2.45		16.2	2.13		Tbath=31.5C
	25	12.2	97.8			2.5			2	
	25	12.5	98.1	15.2	2.45		17.4	2.13		
	25	13.3	98.9			0.8			0.7	Tbath=30.5C
	25	13.4	99.0	15.2	2.45		17.25	2.13		
	25	14.2	99.8	15	2.45		16.9	2.13		Tbath=31C
	25	14.3	99.9			0.8			0.7	
	25	14.7	100.3	15.1	2.45		17.1	2.13		Tbath=31C
	25	15.2	100.8	14.9	2.45		16.6	2.13		Tbath=32C
	25	15.3	100.9		0.1			0.1		current reduced
	28	7.6	165.2	3.4	0.1		3.8	0.1		Tbath=23C
	28	7.6	165.2			1.7			1.8	turned on stirrer
	28	7.8	165.4							
	28	7.9	165.5		2.45			2.13		current increased
	28	8.1	165.7		2.35					C1 current adjusted
	28	8.3	165.9	16.8	2.35		18.4	2.13		
	28	9.0	166.6			0.8			0.7	
	28	9.4	167.0	16.1	2.35		17.7	2.13		Tbath=29C
	28	10.0	167.6			0.8			0.7	Tbath=30C
	28	10.2	167.8	16.1	2.35		17.7	2.13		
	28	11.8	169.4			1.4			1.3	
	28	12.0	169.6	15.4	2.35		17.2	2.13		
	28	12.1	169.7	15.5	2.37		17.1	2.13		
	28	12.7	170.4	15	2.37		16.7	2.13		Tbath=37C
	28	13.3	170.9			0.8			0.7	.05mL(1gThioU/20mLD2O) to C2
	28	13.3	170.9							reversed current in C1
	28	14.0	171.6	13.2	2.37		17.3	2.13		
	28	14.5	172.1			1			1.4	
	28	15.0	172.6	13	2.37		18.8	2.13		Tbath=38C
	28	15.3	172.9			0.8			2.3	
	28	15.4	173.0		0.23					reduced current in C1 to 0.23A
	28	15.4	173.0							
	28	21.6	179.2				24.6	2.13	10.5	Tbath=40C,C2 electrode exposed
	28	21.8	179.4	3.4	0.23		23.55	2.13		
	29	7.7	189.3			1.3			11	
	29	7.9	189.5							
	29	8.5	190.1	4	0.23		27.8	2.13		
	29	8.5	190.1							C2 electrolyte replaced
	29	9.2	190.8					2.13		C2 started, cooling coil added
	29	9.3	190.9							recombined D2O emptied
	29	10.1	191.7							started flow in cooling coil
	29	10.3	191.9	4	0.23		11.1	2.13		Tbath=24C
	29	11.2	192.8	4.2	0.23		11.5	2.13		Tbath=20C
	29	11.3	192.9						1.4	
	29	12.8	194.4	4.1	0.23		11.6	2.13		Tbath=20C
	29	13.3	194.9						1.4	
	29	14.0	195.6	4.1	0.23		11.7	2.13		Tbath=20.5C
	29	15.2	196.8						1.4	
	29	15.5	197.1							recorder paper changed
	29	21.5	203.1	4.1	0.23		10.5	2.13	6	Tbath=20.5
	29	21.6	203.2				12.4	2.13		
	30	7.5	213.1	4.1	0.23		10	2.13		Tbath=20.5C
	30	7.6	213.2						7	
	30	7.8	213.4			2.3				
	30	7.9	213.5		2.3					
	30	8.7	214.3							.05mL(1gThioU/20mLD2O) to C2
	30	8.8	214.4							collected recombined D2O
	30	9.0	214.6			0.8				
	30	9.7	215.3	14.1	2.3		13.2	2.13		Tbath=21.5C

Table C2. Detailed Operaton History of Cells 1B and 2B (Cont'd)

Month	Date	Hour	Run time	Cell#1 Voltage (V)	Current (A)	H2O (mL)	Cell#2 Voltage (V)	Current (A)	D2O (mL)	Comments
	30	9.8	215.4			0.9			0.7	
	30	10.7	216.3	14	2.3		13.2	2.13		
	30	10.8	216.4			0.8			0.7	
	30	11.8	217.4	14	2.3		13.1	2.13		Tbath=21.5C
	30	11.8	217.4			0.8			0.7	
	30	12.7	218.3	13.9	2.3		13.2	2.13		Tbath=21.5C
	30	12.8	218.4			0.8			0.7	
	30	13.7	219.3	13.9	2.3		13.2	2.13		
	30	14.0	219.6							reversed polarity in C1
	30	14.7	220.3			3.8				
	30	14.7	220.3						2.1	
	30	15.0	220.6	14.3	2.3		14	2.13		Tbath=21.5C
	30	21.3	226.9			6	12.2	2.13	7	collected recombiner D2O
	30	21.4	227.0	14.95	2.305		15.45	2.13		Tbath=21C
	31	7.5	237.1	1.8	2.3		12.3	2.13		C1 electrode exposed(1/5)
	31	7.6	237.2			9				
	31	7.8	237.4						8.5	(1mL .1M LiOD to C2)
	31	8.0	237.6							recombined D2O collected
	31	8.7	238.3	15.8	2.3		15.9	2.3		Tbath=19C
	31	8.8	238.4			0.8			0.8	
	31	9.4	239.0	15.9	2.3		16	2.3		Tbath=19C
	31	9.9	239.5			0.8			0.8	
	31	11.0	240.6	15.7	2.3		16	2.3		
	31	11.0	240.6			0.8			0.8	
	31	11.7	241.3	15.9	2.3		16.2	2.3		
	31	11.7	241.3					2.13		C2 current reduced
	31	11.9	241.5			1.5			2	(1.2mL 0.1M LiOH to C1)
	31	12.0	241.6		2.37					
	31	13.4	243.0	14.7	2.37		16.3	2.13		Tbath=18C
	31	14.4	244.0	14.4	2.37		16	2.13		Tbath=18C
	31	14.8	244.4						2.2	
	31	14.9	244.5		2.5					
	31	15.9	245.5	14.2	2.5		16.9	2.13		Tbath=17.5C
	31	16.2	245.8			0.3			1.2	
	31	16.3	245.9	14.6	2.6		17.5	2.14		
	31	16.8	246.4		0.26				0.21	
	31	16.8	246.4							recombined D2O collected
Sept.	1	7.7	261.3	4	0.26		4.8	0.21		Tbath=16C
	1	7.8	261.4		2.35			2.13		
	1	8.0	261.6			2.3			2.2	(2.2mL 0.1MLiOD to C2)
	1	8.2	261.8	14.2	2.35		15.4	2.13		
	1	8.6	262.2	13.9	2.35		15.1	2.13		
	1	9.1	262.7			0.7			0.6	
	1	9.1	262.7	14	2.35		15.2	2.13		
	1	9.7	263.3	13.7	2.35		15	2.13		Tbath=18C
	1	10.1	263.7			0.8			0.6	
	1	10.2	263.8		2.33					
	1	11.0	264.6	13.6	2.33		14.9	2.13		
	1	11.0	264.6		2.3					
	1	11.0	264.6			1			1	(1mL 0.1M LiOD to C2)
	1	12.1	265.7	13.6	2.3		14.7	2.3		
	1	12.1	265.7			1			1.2	(1.2mL 0.1M LiOD to C2)
	1	12.1	265.7			0.2				
	1	12.2	265.8			0.3				
	1	13.1	266.7	14	2.3		14.1	2.3		Tbath=17C
	1	14.1	267.7			0.8			0.8	
	1	15.0	268.6	14.1	2.3		14.2	2.3		Tbath=16.5C
	1	15.1	268.7			0.8			0.8	
	1	15.2	268.8	14.6	2.3		14.6	2.3		

Table C2. Detailed Operaton History of Cells 1B and 2B (Cont'd)

Month	Date	Hour	Run time	Cell#1 Voltage (V)	Current (A)	H2O (mL)	Cell#2 Voltage (V)	Current (A)	D2O (mL)	Comments
	1	15.2	268.8		0.15			0.15		
	1	15.3	268.9			3			3	
	1	15.4	269.0							C1 polarity reversed
	5	7.6	357.2	3.9	0.15		3.6	0.15		Tbath=14C
	5	7.8	357.4			2.2			2.2	
	5	8.2	357.8							recombined D2O collected
	5	9.1	358.7		0	D2O				C1 off, changed to 1M LiOD
	5	11.0	360.6		0.23			0.21		
	5	11.8	361.4	2.5	0.23		4.2	0.21		
	5	14.5	364.1	2.6	0.23		4.1	0.21		Tbath=17.5C
	6	7.6	381.2	2.6	0.23		4	0.21		Tbath=19C
	6	7.7	381.3			1.4			1.7	
	6	12.8	386.4	2.7	0.23		4.1	0.21		Tbath=19C
	6	14.8	388.4	2.7	0.23		4.1	0.21		Tbath=19C
	7	8.0	405.6							C1 connected to recombiner
	7	8.1	405.7	2.6	0.23		4	0.21		Tbath=19.4C
	7	8.2	405.8						1.5	
	7	8.3	405.9	5.2	2.3	3				
	7	9.5	407.1			1				
	7	11.3	408.9			1				Tbath=20.5C
	7	11.9	409.5			1				
	7	13.0	410.6	5.1	2.3	1	4	0.21		Tbath=20.8C
	7	14.2	411.8			1				
	7	15.0	412.6			1.5				
	7	21.3	418.9	5.2	2.3					
	8	7.5	429.1	4.8	2.3		3.9	0.21		Tbath=21C
	8	7.5	429.1			6			3	
	8	8.9	430.5			0.5				autofeed:40%&2
	8	9.0	430.6							recombined D2O removed
	8	9.9	431.5							autofeed:35%&2
	8	11.9	433.5							autofeed:30%&2
	8	12.9	434.5							autofeed:25%&2
	8	14.7	436.3	5.2	2.3		4.1	0.21		Tbath=22.5C
	8	14.9	436.5						1.5	autofeed:27%&2
	8	15.0	436.6							flow in coil increased
	9	11.0	456.6	5.2	2.3		4.4	0.21	1.5	autofeed:30%&2
	9	11.3	456.9	5.7	2.3		4.5	0.21		
	10	13.0	482.6				4.4	0.21		autofeed:100%&120 for 3 minute
	10	13.0	482.6	6	2.3					autofeed:35%&2
	10	13.1	482.7	5.8	2.3					
	11	7.5	501.1	6	2.3		4.3	0.21	1.5	Tbath=17C, autofeed off
	11	7.7	501.3							autofeed:35%&2
	11	11.8	505.4							recombiner sample taken
	11	13.5	507.1							.1mL(1gThioU/20mL) added to C1
	11	15.3	508.9						1	
	12	7.7	525.3	5.7	2.3		4.3	0.21	1	autofeed:100%&120 for 2 min
	12	7.8	525.4							recombined D2O from C1 taken
	12	9.8	527.4							autofeed stopped
	12	11.9	529.5							autofeed started
	12	13.4	531.0							.1mL(1gThioU/20mL) toC1
	12	13.5	531.1				16.1	2.1		.1mL(1g/20mL)to C2 & 35%&2
	12	13.6	531.2							C1 pump stopped
	12	15.1	532.7	6.1	2.3		16.1	2.1	1	C1 pump on, C2:40%&2
	12	16.1	533.7					0.21		C2 pump lost prime
	13	7.6	549.2	6.4	2.3		4.9	0.21	2.2	C1 pump stopped, Tbath=18C
	13	7.7	549.3							C1 recombined D2O taken
	13	8.1	549.7							recombiner to C2
	13	9.9	551.5						2	C2 recombined D2O taken
	13	11.4	553.0				20.5	2.1		C2:40%&2

Table C2. Detailed Operaton History of Cells 1B and 2B (Cont'd)

Month	Date	Hour	Run time	Cell#1 Voltage (V)	Current (A)	H2O (mL)	Cell#2 Voltage (V)	Current (A)	D2O (mL)	Comments
	13	11.5	553.1							C1:35%&2
	13	13.0	554.6	6.1	2.3		18.3	2.1		C2:35%&2, Tbath=18.8C
	13	15.1	556.7	6.2	2.3		18.7	2.1		
	14	7.6	573.2	5.9	2.3		19.9	2.1		Tbath=19C
	14	7.6	573.2			3				
	14	7.7	573.3							C2 recombined D2O taken
	14	11.9	577.5							0.001g FeSO4.7H2O added-C1&C2
	14	14.6	580.2	6.3	2.3		19.5	2.1		Tbath=19C
	14	15.2	580.8							D2O added to both cells
	15	7.5	597.1	29.6	0		19.4	2.1		C1 empty, C2 .5" electrolyte
	15	7.7	597.3	6.5	2.3		27.2	2.1		C2 recombined D2O removed
	15	8.1	597.7							C1 & C2:4
	15	8.6	598.2							C1:2
	15	9.6	599.2	6.4	2.3		26.7	2.1		C1 P off, C2:6
	15	10.5	600.1							C1:35%&2
	15	13.0	602.6	6.3	2.3		28.1	2.1		Tbath=19.8C, C1:off
	15	14.0	603.6	6.3	2.3		30.1	1.67		C2 pump off
	15	15.0	604.6	2.7	0.23		7.4	0.21		filled C1&2
	18	7.5	669.1	2.5	0.23		9.6	0.21		Tbath=19C
	18	7.6	669.2							C2 re-D2O taken, switch to C1
	18	7.8	669.4			x			x	C1&2:.1mL(1gThioU/20mL) added
	18	7.9	669.5	6.5	2.3		30	1.71		C2:1.8mL .1M LiOD added
	18	8.0	669.6							C1:35%&2
	18	10.8	672.4				22	1.6		C1 P off,C2:1.2mL.1MLiOD added
	18	11.7	673.3	6.4	2.3		21.3	1.6		
	18	13.1	674.7							C1 pump on
	18	14.1	675.7				13.5	2.1		C2:1mL 1M LiOD added
	18	15.0	676.6	6.4	2.3		13.6	2.1		Tbath:19.5
	19	7.5	693.1	7.2	2.3		16.6	2.1		Tbath=20C, C1&2 pumps off
	19	11.3	696.9	7.2	2.3		15.7	2.1		Tbath=19.8C
	19	12.9	698.5	6.9	2.3		14.8	2.1		.001g Cr2(SO4)3 to C1&2
	19	14.4	700.0	6.9	2.3		14.8	2.1		Tbath=19.7, C1&2:35%&2
	20	7.6	717.2	7	2.3		14.4	2.1		Tbath=19.8C
	20	7.8	717.4							C2 sample taken
	20	9.0	718.6	7.5	2.3		20.4	2.1		
	20	10.3	719.9							C1 pump off
	20	11.8	721.4							C1 pump on
	20	13.8	723.4	7.6	2.3		22.6	2.1		Tbath=20C, C1 pump off
	20	14.3	723.9							C1&2:35%&2
	20	22.8	732.4							C2 cell popped
	21	7.9	741.5	8.2	2.3		29	1.4		C1 pump off, Tbath=19.8C
	21	8.1	741.7							C1 sample taken
	21	8.4	742.0							C2:1mL 1MLiOD added
	21	8.5	742.1							recombiner to C2
	21	10.3	743.9	8	2.3		13.4	2.1		
	21	13.0	746.6	7.8	2.3		13.5	2.1		Tbath=20C
	21	15.1	748.7	7.6	2.3		13.4	2.1		Tbath=20C, C1:35%&2
	22	7.6	765.2	7.4	2.3		13.4	2.1		Tbath=20C
	22	7.8	765.4							C2 sample taken switched to C1
	22	7.9	765.5							C1&2:.1ml(1gThioU/20mL) added
	22	9.2	766.8	8.1	2.3		17.3	2.1		
	22	10.9	768.5	8	2.3		17.5	2.1		
	22	13.7	771.3	7.5	2.3		18	2.1		
	22	15.1	772.7	7.7	2.3		18.5	2.1		Tbath=20C, C1 sample taken
	22	15.1	772.7							all cell off

Figure C1. Cell 1A Current Settings

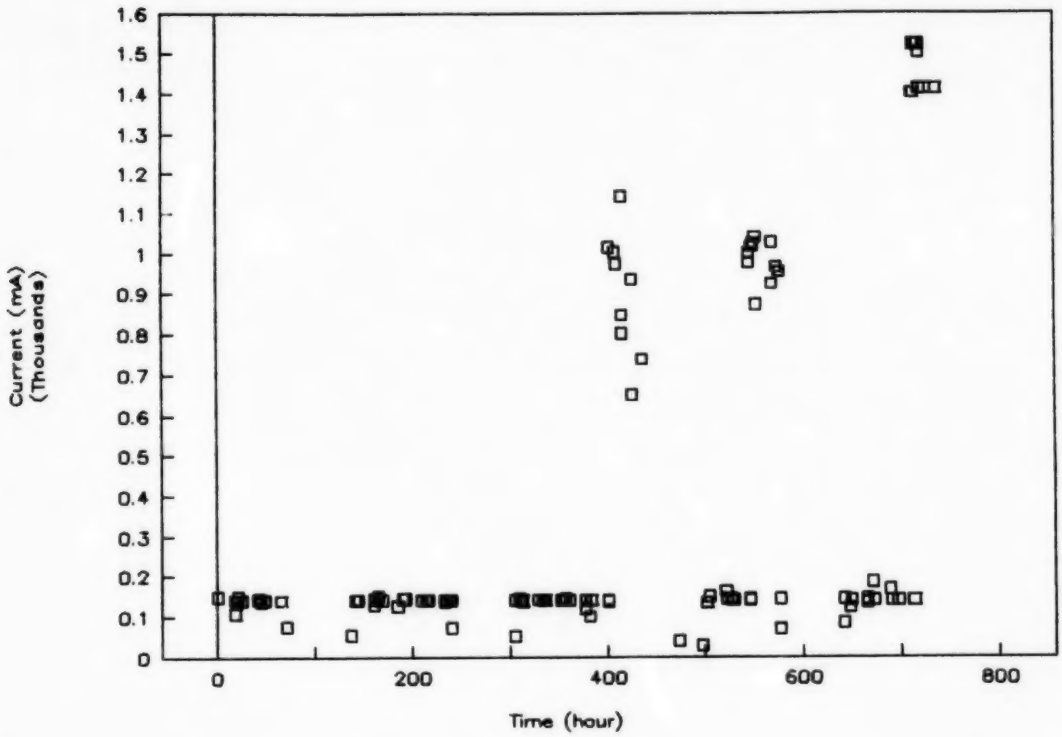
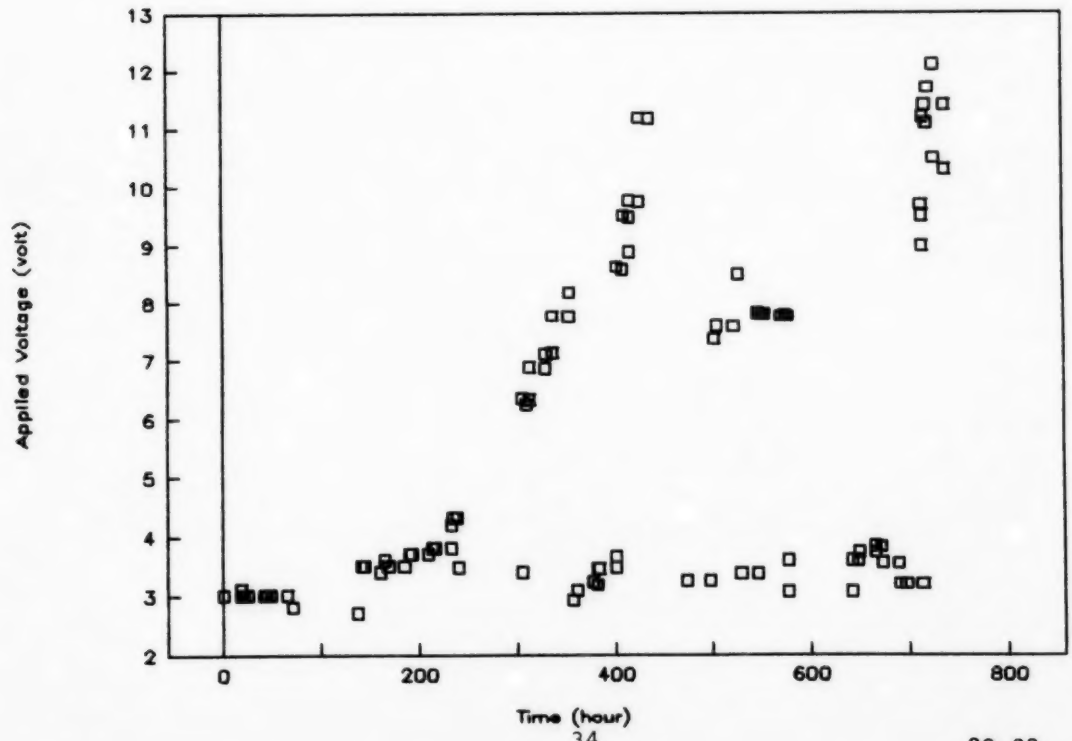


Figure C2. Cell 1A Voltage Settings



90-28

Figure C3. Cell 2A Current Settings

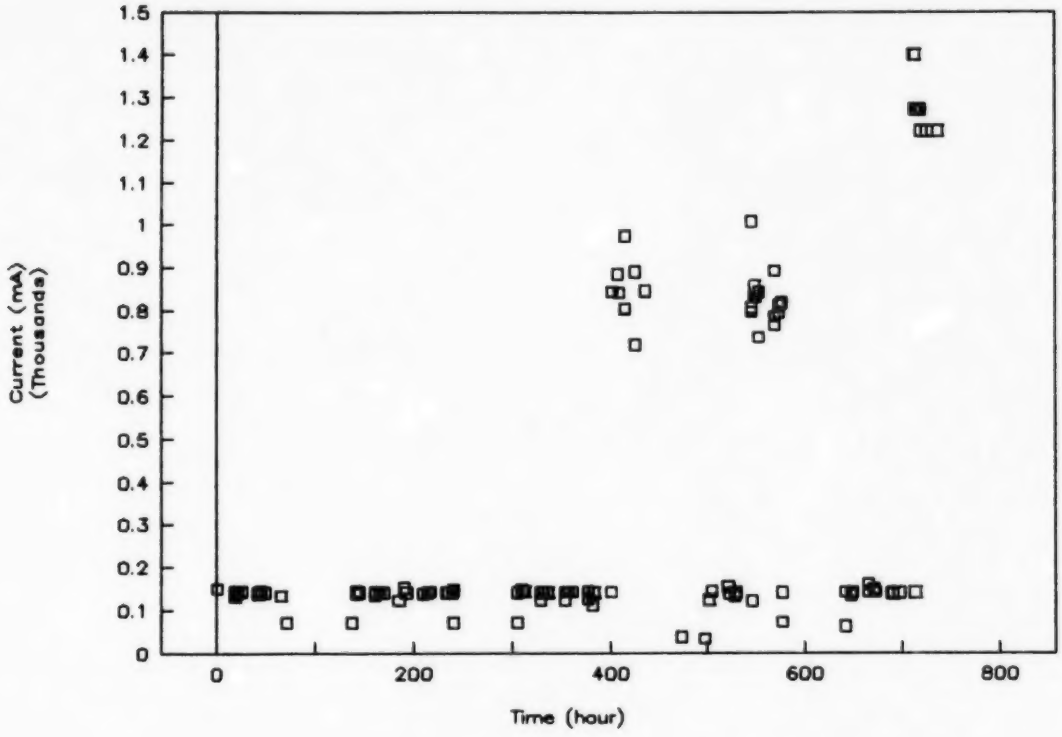
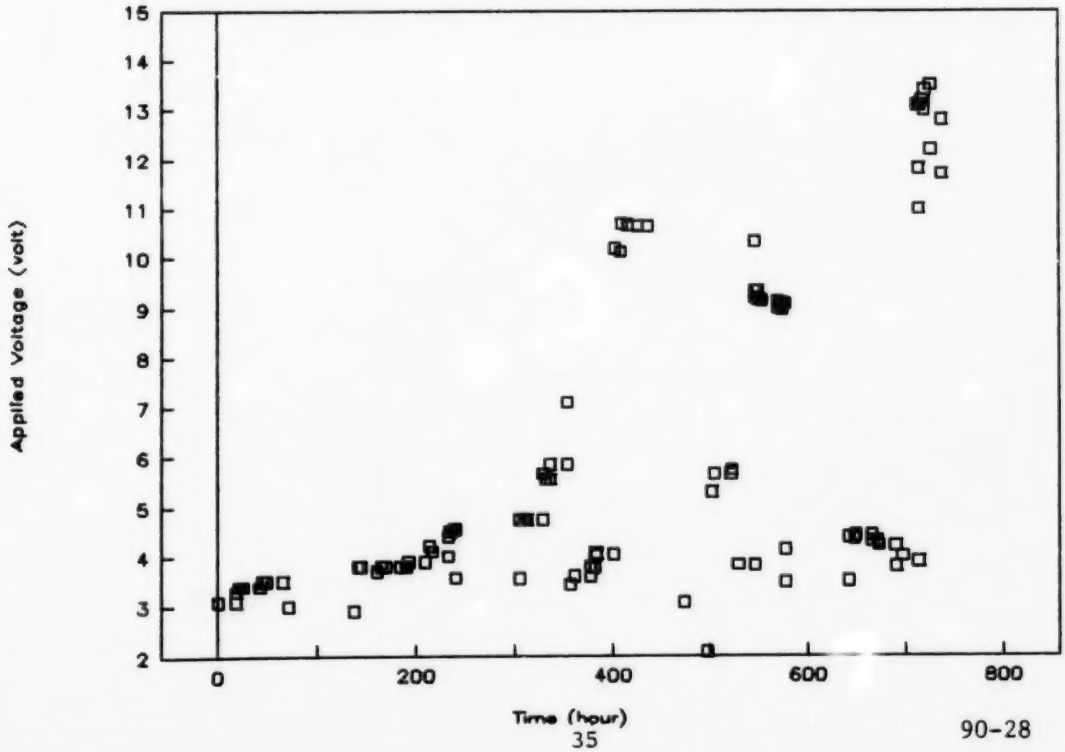


Figure C4. Cell 2A Voltage Settings



90-28

Figure C5. Cell 1B Current Settings

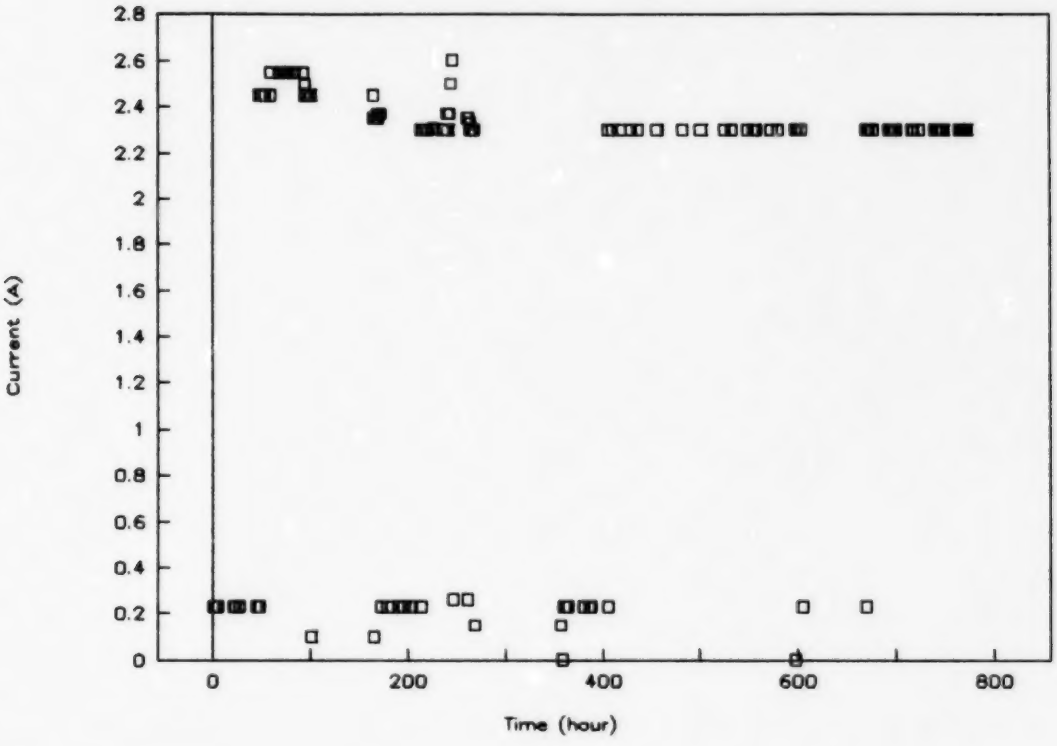


Figure C6. Cell 1B Voltage Settings

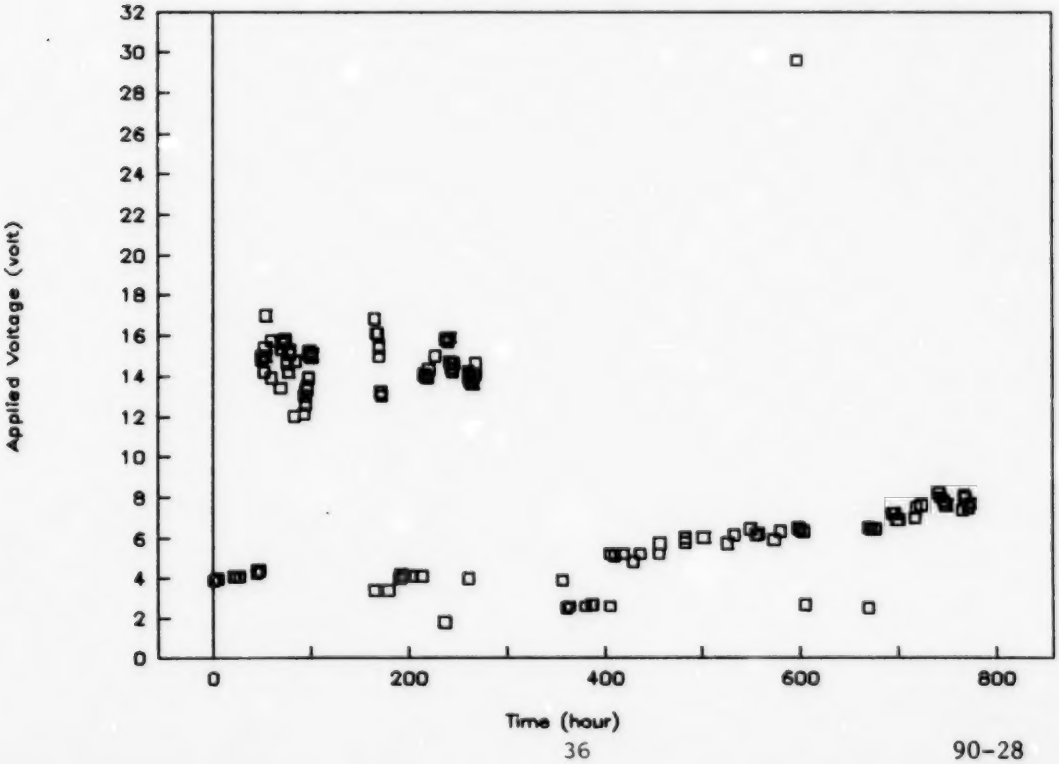


Figure C7. Cell 2B Current Settings

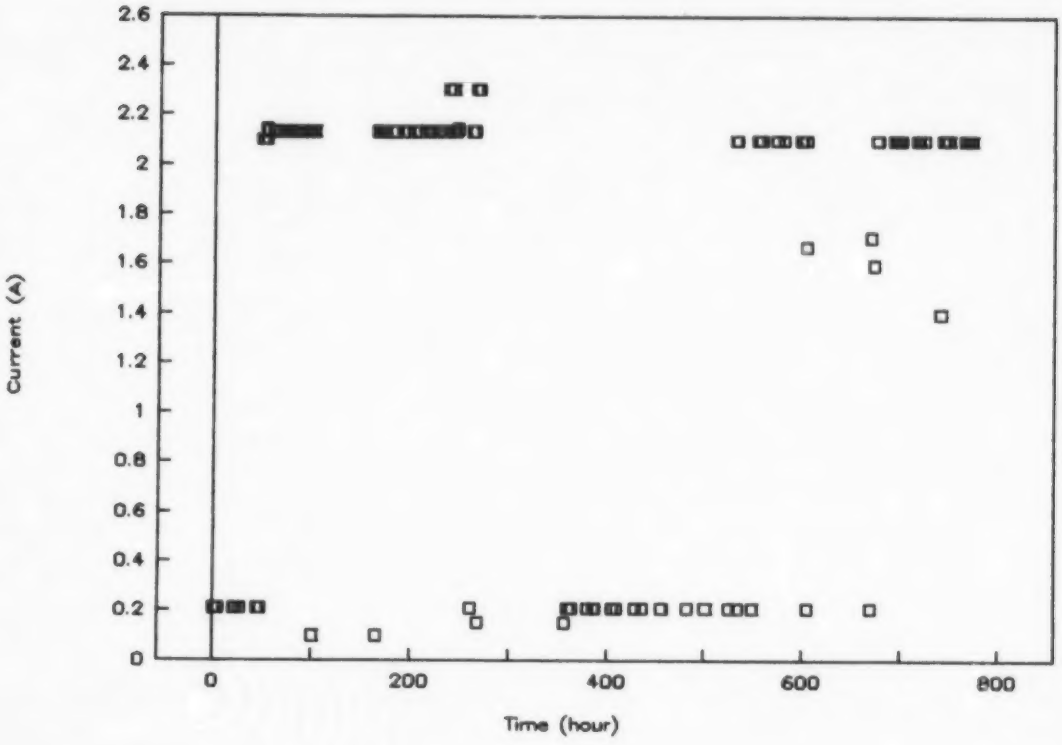


Figure C8. Cell 2B Voltage Settings